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(54) Title: SURFACE TREATMENT

(57) Abstract: This invention describes a surface treatment composition, preferably for personal use, comprising at least one surfactant and at least two different organic acids and/or salts or organic acids, and wherein the total concentration of the organic acids and/or salts in the composition is at least 0.5 % (w/v). The invention further extends to a method of treating a surface, preferably hair or skin, to remove dirt and to remove or inhibit microbial growth. The acids are preferable citric and lactic acid, and preferably act to buffer the composition together with their salts.

SURFACE TREATMENT

This invention relates to surface treatment compositions, and methods of treating surfaces. The invention relates particularly, but not exclusively, to compositions for treatment of the skin or hair to effect cleaning, and to related methods. For the purpose of this specification the term "cleaning" denotes removal of dirt (including grease) and/or combating of microorganisms.

Many compositions used for surface treatment include a surfactant or combination of surfactants which help to release dirt and/or microorganisms from the hard surface. Such compositions may also contain biocidal agents. Such compositions must be formulated carefully. For example surfactants may denature biocidal agents. Many biocidal agents have a detectable odour and the type and amount of a fragrance - typically an expensive ingredient - may need to be carefully selected. Foaming qualities are usually desired and these can be compromised by subtle aspects of the formulation selection. The viscosity of the compositions can be hard to control. The desired viscosity may be reduced as a function of time, temperature or pH. Optimal action of a biocidal agent in the composition may require a particular pH range, whereas a different pH range may be needed for optimal viscosity.

The nature and combination of surfactants chosen will also contribute significantly to the physical properties of such a formulation. Consideration of the solubilisation of biocidal agents in such systems is crucial, to obtain and maintain efficacy.

Frequently, a compromise is reached in known compositions in which either the viscosity or biocidal efficacy of the composition, or both, is sacrificed in part, in order to provide a composition which provides at least reasonable viscosity and biocidal activity. In many known compositions, the pH of the composition is raised or lowered in order to optimise the viscosity, or a combination of surfactants is utilised to promote thermodynamic instability, in order to increase efficacy. In some compositions where viscosity is not compromised, biocidal activity is less than optimal.

It would therefore be advantageous to provide a surface treatment composition which provides a surfactant and biocidal action at optimal viscosity for good handling and high surfactant efficacy, but without reducing the efficacy of any biocidal agent(s) in the composition. It would also be advantageous to provide a composition which can be stored for relatively long periods of time without significant detrimental alteration of viscosity of the composition, or significant reduction in either surfactant effect or biocidal activity. Furthermore, it would be desirable to provide a surface treatment composition which includes a surfactant, the composition being at the optimum pH for good viscosity and viscosity maintenance, and good surfactant and biocidal action.

It is therefore an aim of preferred embodiments of the invention to overcome the problems of the prior art.

According to a first aspect of the present invention, there is provided a surface treatment composition comprising at least one surfactant and at least two

compounds selected from organic acids and salts of organic acids, and wherein the total concentration of the organic acids and salts of organic acids in the composition is at least 0.5% (w/v).

Preferred acids for use in the present invention are carboxylic acids. Preferred salts of organic acids for use with the present invention are carboxylates, preferably alkali metal carboxylates, more preferably potassium or, especially, sodium salts.

Preferred carboxylic acids/carboxylates are aliphatic; especially saturated aliphatic.

Other suitable organic acids and salts thereof may include benzene sulphonic acid and other aromatic sulphonic acids, uric acid and other purine-containing acids and ascorbic acid and other sugar-derived acids.

Suitably one of the organic acids or salt of an organic acid has two or more carboxylic acid or carboxylate groups.

In a preferred embodiment, one of said compounds is an organic acid or a salt of an organic acid, having two or more carboxylic acid or carboxylate groups, and another of said compounds is an organic acid or a salt of an organic acid, having one carboxylic acid or carboxylate group.

In another preferred embodiment, one of said compounds is an organic acid or a salt of an organic acid, having three carboxylic acid or carboxylate groups, and another of said

compounds is an organic acid or a salt of an organic acid, having one or two carboxylic acid or carboxylate groups.

Suitable organic acids/salts with one carboxylic acid or carboxylate group include linear or branched optionally substituted hydroxyalkyl carboxylic acids/salts or alkylmonocarboxylic acids/salts, of 1 to 8 chain carbon atoms, preferably 1 to 6 chain carbon atoms.

A suitable monocarboxylic acid/salt is formic, acetic, chloroacetic, dichloroacetic, benzoic, 2,4,6-trihydroxybenzoic, 2-aminobenzoic, pyruvic, quinolinic, 2-chlorobenzoic, glyoxylic, thioacetic, glyceric, acetoacetic, hippuric, glycolic acid, and especially, lactic acid; or salts thereof.

Suitable organic acids/salts with two carboxylic acids or carboxylate groups include linear or branched optionally substituted hydroxyalkyldicarboxylic acids/salts or alkyldicarboxylic acids/salts, of 2 to 10 chain carbon atoms, preferably 2 to 8 chain carbon atoms. Preferred organic dicarboxylic acids/salts include tartaric, oxalic, maleic, aspartic, L-glutamic, oxaloacetic, 2-oxogluteric, malonic, phthalic, methylmalonic, mesaconic, methylsuccinic, glutaric, malic and adipic acids or salts thereof.

Suitable organic acids/salts with three carboxylic acids or carboxylate groups include linear or branched optionally substituted hydroxyalkyltricarboxylic acids/salts, alkyltricarboxylic acids/salts, of 3 to 10 chain carbon atoms, preferably 3 to 8 chain carbon atoms. Preferred organic tricarboxylic acids/salts include L-

argininosuccinic, isocitric and, especially, citric acid or salts thereof.

In a particularly preferred embodiment of the invention the composition comprises two organic acids and/or salts of organic acids; preferably carboxylic acids/salts. Preferably one of the organic acids/salts comprises lactic acid and/or lactate and another of the organic acids/salts comprises citric acid and/or citrate.

Preferably the composition comprises both a first organic acid and a salt of that organic acid.

Preferably the composition comprises both a second organic acid and a salt of that organic acid.

Suitably the total concentration of all of the organic acids and/or salts thereof in the composition is at least 1% (w/v), preferably at least 2% (w/v).

Suitably the total concentration of both organic acids and/or salts thereof in the composition is no more than 10% (w/v), preferably no more than 7.5% (w/v).

Suitably the pH of the composition is no more than 6, preferably no more than 5.5, more preferably no more than 5 and most preferably no more than 4.8.

Suitably the pH of the composition is at least 2, preferably at least 3 and more preferably at least 4.

One or, preferably, all of the organic acids and/or salts preferably act to buffer the composition to a desired pH.

The surfactant may be anionic, cationic, non-ionic, zwitterionic or amphoteric.

There may be more than one surfactant, preferably being independently selected from an anionic, cationic, non-ionic, zwitterionic or amphoteric surfactant.

Suitable non-ionic surfactants include alkoxylated alcohols, particularly alkoxylated fatty alcohols. These include ethoxylated and propoxylated fatty alcohols, as well as ethoxylated and propoxylated alkyl phenols, both having alkyl groups of from 7 to 16, more preferably 8 to 13 carbon chains in length.

Examples of alkoxylated alcohols include certain ethoxylated alcohol compositions presently commercially available from the Shell Oil Company (Houston, TX) under the general trade name NEODOL (trade mark), which are described as linear alcohol ethoxylates, certain compositions presently commercially available from the Union Carbide Company, (Danbury, CT) under the general trade name TERGITOL (trade mark) which are described as secondary alcohol ethoxylates, and contain compositions present commercially available from Clariant (UK) under the general trade name GENAPOL (trade mark) and which are described to be linear and branched alcohol ethoxylates.

Examples of alkoxylated alkyl phenols include certain compositions presently commercially available from the Rhône-Poulenc Company (Cranbury, NJ) under the general trade name IGEPAL (trade mark), which are described as octyl and nonyl phenols.

Suitable anionic surfactants include linear C_8 to C_{16} alkyl sulphates, C_8 to C_{16} alkylsulphonates, C_8 to C_{16} alkylbenzenesulphonates, C_8 to C_{16} alkyldiphenyloxide disulphonates and C4 to C_{16} alkylated naphthalene sulphonates. Suitable examples of anionic surfactants are sodium lauryl sulphonate and sodium dodecyl benezene sulphonate, or mixtures thereof. Preferably the anionic surfactant is selected from those comprising an alkali metal or ammonium cation.

A preferred composition of the present invention includes an anionic surfactant.

Suitable amphoteric surfactants include betaines.

A preferred composition of the present invention includes an amphoteric surfactant.

An especially preferred composition of the present invention includes an anionic surfactant in combination with an amphoteric surfactant. Preferably the ratio of the weight of the anionic surfactant to the weight of the amphoteric surfactant exceeds 1:1, and more preferably exceeds 2:1. Most preferably it exceeds 4:1. In highly preferred embodiments it exceeds 6:1.

Suitably the total concentration of the surfactant(s) in the composition is at least 2% (w/v), preferably at least 5% (w/v) and more preferably at least 8% (w/v).

Suitably the total concentration of the surfactant(s) in the composition is no more than 25% (w/v), preferably no more than 20% (w/v).

Suitably the composition is an aqueous composition. Preferably the composition comprises at least 50% (w/v) water, more preferably at least 60% (w/v), most preferably at least 70% (w/v).

The composition may comprise one or more further ingredients such as preservatives, thickeners, fragrance, chelating agents, and sodium chloride, for example.

The composition may contain a biocidal agent. The biocidal agent may be a bactericide, a viricide, a fungicide, a parasiticide, herbicide, algicide or any mixture of a combination thereof. Preferably it is a bactericide.

Suitably biocidal agents include phenolic compounds, such as PCMX.

There may be more than one biocidal agent present in the composition.

When a biocidal agent is present it may suitably be at a total concentration in the composition of at least 0.1% (w/v), preferably at least 0.2% (w/v) and more preferably at least 0.4% (w/v). Preferably it is present in an amount of up to 2% (w/v), more preferably up to 1% (w/v), most preferably up to 0.6% (w/v).

However it is believed that in preferred embodiments the acids and/or salts used in the invention may provide biocidal action, and it is possible that a traditional biocidal agent, such as an aromatic or heteroaromatic

compound, notably a phenolic compound (for example PCMX), may not be needed in some embodiments. Accordingly compositions not containing such a biocidal agent are within the scope of the present invention.

Preferred compositions of the present invention have a foaming action with water on the surface to be treated.

According to a second aspect of the present invention there is provided a surface treatment composition comprising at least one surfactant and at least two different organic buffers.

Suitably the organic buffers comprise organic acids and salts thereof, as described above. Of course the buffers are selected to be compatible with each other in the composition, and compatible with other components of the composition.

Suitably the or each surfactant is as described for the first aspect of the invention.

Suitably the composition further comprises a biocidal agent as described for the first aspect of the invention.

Other definitions given above in relation the first aspect are applicable to the second aspect.

The composition of the first or second aspect is preferably a liquid skin cleaner (for example a hand wash), a shower gel, or the like.

In accordance with a third aspect of the present invention there is provided a package containing a composition of the first or second aspect, the package comprising a container for the composition and a restricted dispenser outlet therefrom under the control of a user. The restricted dispenser outlet could be, for example, a spray nozzle of a pressurised canister, or the outlet of a pump-action container, for example a press-action "tap" or the spray nozzle of a trigger spray container.

According to a fourth aspect of the invention there is provided a method of treating a surface, the method comprising the step of contacting the surface with the surface treatment composition of the first or second aspects of the invention.

Suitably the surface is a surface of a person, in particular the skin or hair of a person.

The method may comprise coating the surface with the composition, directly from a container or via the agency of a separate part, for example a sponge, cloth or the hand, or spraying the surface with the composition.

The method may comprise the final step of rinsing the surface with an aqueous media, suitably clean water.

Example

The various aspects of the invention will now be described with reference to the following non-limiting examples in which the following materials are used:

PCMX - parachloro meta-xylenol, supplied by Thomas Swan, Durham

EMPICOL ESB 70 (SLES) - sodium lauryl (C_{12-16}) ethoxy (2-3 EO) - sulphate surfactant, supplied by Huntsman

EMPIGEN BSFA - a betaine amphoteric surfactant, supplied by Huntsman

KATHON CG - a preservative; a mixture of thiazolinones, supplied by Rohn & Haas

JAGUAR EXCEL - a guar gum supplied by Rhodia

Pine fragrance

EMPICOL XPE/H - pearliser, supplied by Huntsman

EMPICOL, EMPIGEN, KATHON and JAGUAR EXCEL are trade marks.

A composition of the invention was made up according to Formulation A given in Table 1 below, in which lactic acid/sodium lactate and citric acid/sodium citrate were used as two different buffering agents. A second formulation, Formulation B was also prepared in which the citric acid/sodium citrate was omitted. A control formulation, Formulation C was prepared in which no such organic acid or salt was present.

Table 1

Ingredient	Concentration (%w/v)		
	Formulation A	Formulation B	Formulation C
PCMX	0.5	0.5	0.5
EMPICOL ESB 70	9.0	9.0	9.0
EMPIGEN BSFA	1.5	1.5	1.5
Lactic acid	To pH 4.7	To pH 4.7	-
Sodium lactate	1.0	1.0	-
EMPICOL XPE/H	1.5	1.5	1.5
Fragrance	0.2	0.2	0.2
KATHON CG	0.02	0.02	0.02
Tetrasodium EDTA	0.3	0.3	0.3
Sodium citrate	0.7	-	-
Citric acid	To pH 4.7	-	-
JAGUAR EXCEL	0.3	0.3	0.3
Sodium chloride	-	-	Q.S
Deionised water	to 100%	to 100%	to 100%

The anti-microbial efficacy of Formulation A, Formulation B and Formulation C against *Staphylococcus aureus* (NCTC 10788) and *Escherichia coli* (NCTC 10418), was tested by performing a Handwash Efficacy Suspension Test.

The Handwash Efficacy Suspension Test is based on a standard test for the assessment of the rapid germicidal activity for antibacterial liquid and bar soap products, test prEN12054 - chemical disinfection and antiseptics - Products for hygienic and surgical handrub and handwash, bactericidal activity, test method and requirements (phase 2, step 1); British Standard Institute draft for public

comment 95/561926 July 1995; but with use of a different *E. coli* strain).

The microbiocidal effect (ME) due to the action of the composition over the test contact time at the temperature at which the test was performed is expressed by the formula:

$$ME = \text{Log } N_C - \text{Log } N_D$$

where:

N_C = Number of cfu/ml of the relevant control test (test mixture without composition).

N_D = Number of cfu/ml of the test mixture after the action of the composition.

Results are graded as follows:

ME values obtained	Activity
>4.0	Excellent
3.0 - 4.0	Good
1.5 - 3.0	Moderate
0.5 - 1.5	Poor
<0.5	No activity

Formulations A, B and C were diluted in hard water to give a 50% v/v concentration, and were tested against *S.aureus* by contacting Formulations A, B and C with *S.aureus* cultures for one minute.

The tests were repeated a further two times to give a total of three repeats.

The results of the anti-microbial efficacy test against *S.aureus* are shown in Table 2.

Table 2

Formulation	Replicate ME Values			Median ME Value
	Run 1	Run 2	Run 3	
A	2.68	2.48	3.08	**2.68**
B	0.95	0.78	2.23	**0.95**
C	1.38	1.25	1.61	**1.38**

The anti-microbial efficacy of Formulations A, B and C was tested against *Escherichia coli* , using the Handwash Efficacy Suspension Test as detailed above. The results of the test against *E. coli*) is shown in Table 3 below.

Table 3

Formulation	Replicate ME Values			Median ME Value
	Run 1	Run 2	Run 3	
A	4.68	4.95	4.57	**4.68**
B	2.24	2.60	2.60	**2.60**
C	0.24	0.09	0.17	**0.17**

The results of the test as shown in Tables 2 and 3 show that Formulation A exhibited moderate activity against *S.aureus* with a median ME value of 2.68.

Formulation A also showed good activity against *E. coli* achieving a median ME value of 4.68. Formulation B showed moderate activity against *E. coli* compared to Formulation A, with median ME value of 2.6, whereas Formulation C without organic buffers showed no activity against this organism.

Viscosity stability issues were also studied. The polymeric thickener CROTHIX was found to be unstable at the pH of Formulation B (pH 4.7). The pH of the formulation was increased to pH 5.2 to avoid viscosity degradation over time. The microbial efficacy of the product decreased considerably as compared to Formulation B at pH 4.7. Also, after storage at 50°C for two weeks, the pH 5.2 formulation exhibited significant viscosity degradation. It was noticed that pH decreased over this time period. As such, an increase in buffering capacity of Formulation B was investigated by increasing the amount of sodium lactate/lactic acid pairing, which resulted in excess of sodium ions, and as a result, the formulation was not capable of thickening.

Conversely, Formulation A, employing namely sodium citrate/citric acid and sodium lactate/lactic acid, counteracted the loss of thickening capacity at pH 5.2, such that Formulation A at pH 5.2 showed significantly decreased viscosity degradation over time, with no significant loss of biocidal effect, compared to Formulation B in which the pH was increased by 5.2 by addition of further sodium lactate/lactic acid.

The biocidal efficacy of Formulation A at pH 5.2 was much higher (5 log reductions v. *S.aureus* and *E. coli)* compared to Formulation B at pH 5.2.

In further tests of a corresponding composition containing the citric and lactic buffer pairs bur not containing PCMX nor any other accepted biocidal agent, significant biocidal activity was still obtained.

Claims

1. A surface treatment composition comprising at least one surfactant and at least two different organic acids and/or salts or organic acids, and wherein the total concentration of the organic acids and/or salts of organic acids in the composition is at least 0.5% (w/v).

2. A composition as claimed in Claim 1, wherein one of the organic acids or salt of an organic acid has two or more carboxylic acid or carboxylate groups.

3. A composition as claimed in Claim 1 or 2, wherein one of the organic acids or salts of an organic acid has two or more carboxylic acid or carboxylate groups, and another organic acid or salt of an organic acid has one carboxylic acid or carboxylate group.

4. A composition as claimed in any preceding claim, wherein one organic acid or salt has one carboxylic acid or carboxylate group, being a linear or branched optionally substituted hydroxyalkyl carboxylic acid or salt or alkylmonocarboxylic acid or salt comprising 1 to 8 chain carbon atoms.

5. A composition as claimed in any preceding claim, wherein an organic acid or salt with two carboxylic acid or carboxylate groups is a linear or branched optionally substituted hydroxyalkyldicarboxylic acid or salt or alkyldicarboxylic acid or salt, of 2 to 10 chain carbon atoms.

6. A composition as claimed in any preceding claim, wherein the organic acid or salt comprising three carboxylic acids or carboxylate groups is a linear or branched optionally substituted hydroxyalkyltri-carboxylic acid or salt or alkyltricarboxylic acid or salt, of 3 to 10 chain carbon atoms.

7. A composition as claimed in any preceding claim, wherein the composition comprises a first organic acid and a salt of that organic acid and a second organic acid and a salt of that organic acid.

8. A composition as claimed in Claim 7, wherein at least one of the organic acids and salts thereof acts to buffer the composition to a desired pH.

9. A composition as claimed in Claim 7 or 8, wherein the composition comprises lactic acid and/or lactate and citric acid and/or citrate.

10. A composition as claimed in any preceding claim, wherein the total concentration of all of the organic acids and/or salts thereof in the composition is at least 1% (w/v).

11. A composition as claimed in any preceding claim, wherein the pH of the composition is no more than 6.

12. A composition as claimed in any preceding claim, wherein the total concentration of surfactant(s) in the composition is at least 2% (w/v).

13. A composition as claimed in any preceding claim, wherein the composition further comprises a biocidal agent.

14. A surface treatment composition comprising at least one surfactant and at least two different organic buffers.

15. A composition as claimed in Claim 14, wherein the organic acids and salts thereof are as claimed in any one of Claims 2 to 7.

16. A package containing a composition as claimed in any preceding claim, the package comprising a container for the composition and a restricted dispenser outlet under the control of a user.

17. A method of treating a surface, the method comprising the step of contacting the surface with the surface treatment composition as claimed in any of claims 1 to 15.

18. A method as claimed in Claim 17, wherein the method is a method of removing dirt and of removing or inhibiting microbial growth from skin or hair.

19. A composition, package or method substantially as described herein.

INTERNATIONAL SEARCH REPORT

nal Application No
PCT/GB 03/04381

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/06 A61K7/48 A61K7/50 A01N37/02 //(A01N37/02, 37:02,25:30)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A61K A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 262 038 B1 (HEILMAN TIMOTHY J ET AL) 17 July 2001 (2001-07-17) column 1, line 64 -column 2, line 47 column 2, line 59 -column 4, line 30 column 5, line 49 -column 6, line 55	1-6, 10-13, 16-19
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
23 January 2004	04/02/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Donovan-Beermann, T

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51*bis*.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

in relation to this international application, Reckitt Benckiser (UK) Limited is entitled to apply for and be granted a patent by virtue of the following:

An assignment from Andrea Duddington to Reckitt Benckiser (UK) Limited dated 12 November 2003.

An assignment from Paul Frederick Field to Reckitt Benckiser (UK) Limited dated 7 November 2003.

An assignment from David Norman Payne to Reckitt Benckiser (UK) Limited dated 7 November 2003.

This declaration is made for the purposes of all designations.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".